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OCT 10 2002

TC 1700

WITCO CORPORATION
OSI SPECIALTIES GROUP
TARRYTOWN, NEW YORK

SUBJECT

Scale Up of TMS & NEMALA w/ M*M*

DATE

TEST NO. 3196-B

1 Obj: To scale up reaction & produce TT Samples

2
3 Materials: NEMALA 150g (1.51 moles)
4 TMS 220g (1.80 moles)
5 M*M* 0.25 mL (34 ppm)6
7 Equipment: 500 mL 3 neck flask equipped w/ magnetic stirrer,
8 addition funnel & dry ice condenser w/ N₂ inlet.9
10 Procedure: Charge TMS to flask & stir. Charge M*M* & heat
11 to 60°. Charge NEMALA thru addition funnel & maintain
12 Temp < 80°C. Hold @ 90° for 1 hr. & heat to 105°.
13 GC analysis & strip conditions in Comment section14
15 Time Temp Comments

16 8:35 Heating TMS to 60°. Charged M*M*.

17 8:51 67.5 Charging NEMALA w/ addition funnel. About 1 drop/sec.

18 9:02 62.9 Increased feed rate to about 3 drops/sec.

19 * Started adding at 67.5 w/ mantle set on 60°C. Temp dropped
20 to 62.5 before starting to climb. *

21 9:07 64.5

22 9:08 Can see some bubbles forming in flask

23 9:12 65.3

24 9:20 64

25 9:30 60.4 Increased mantle temp to 90°. Hold @ 90° until 10:30

26 9:37 80.1 Addition complete. Still lots of bubbles in flask.

27 or reflux? of TMS (Temp now 92.1.) 9:40

28 9:50 91.8

29 10:00 89.8

30 10:30 89.9 Increased temp to 105°.

31 10:50 103.8

32 11:50 105.7

33 12:30 105.1

DATA BY

Beth A. Gaper

DATE

WITNESSED AND UNDERSTOOD BY

Cathy L. Clegg

DATE

CROSS REFERENCES



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OSI SPECIALTIES GROUP
TARRYTOWN, NEW YORK

9

Cont'd PD-3196-8

DATE:

TEST NO. 3196-9

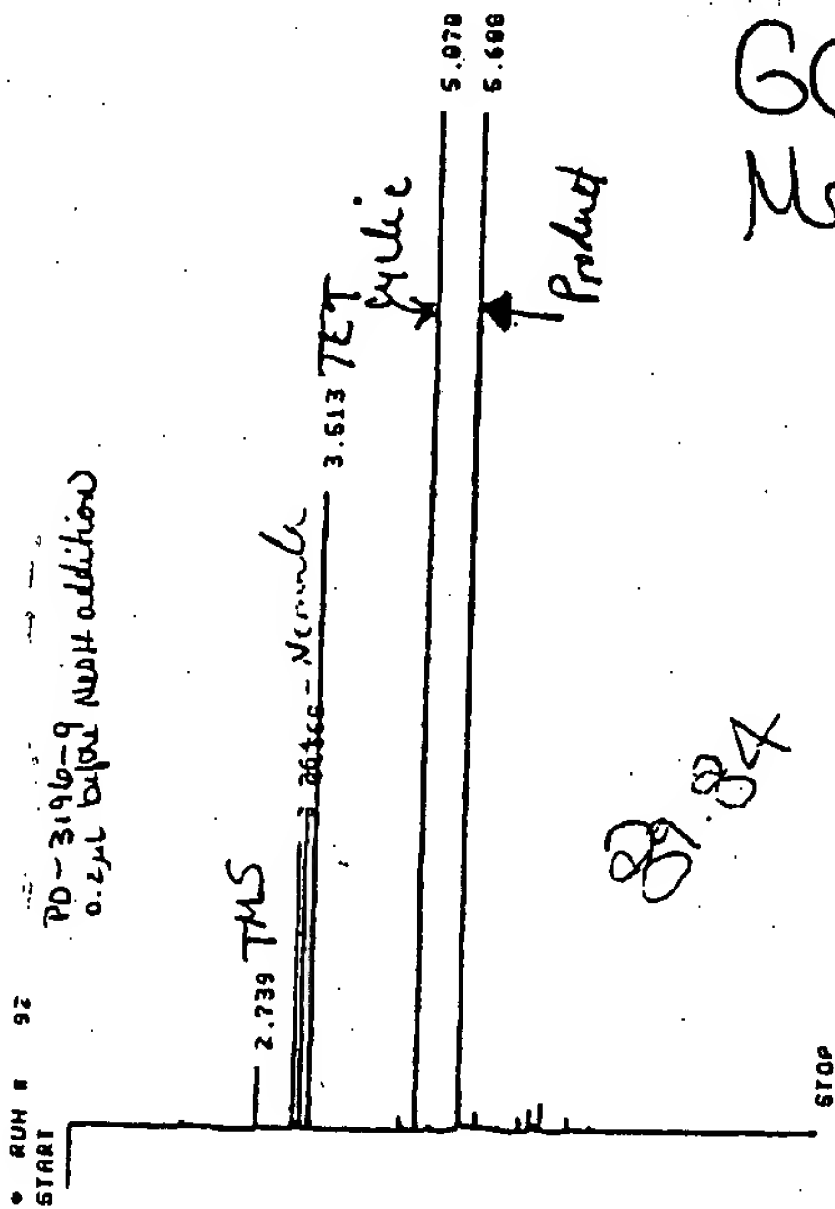
2:00	105.0	1
2:30	104.3	2
3:00	Cooling to Run GC	3
3:40	Charging 65ml MeOH thru addition funnel FD	4
	Conduct cyclic.	5
3:49	Temp @ 26.5 on ice. Heavy foaming w/ addition of MeOH.	6
3:50	All MeOH added.	7

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GC before
MeOH addition



Closing signal file M: SIGNAL .BNC

RUN# 92

METHOD NAME: M:V5669.MET

IDENTIFIER: AGE-3

SIGNAL FILE: M: SIGNAL .BNC

RT	AREA	TYPE	WIDTH	AREA	NAME
2.739	7619	88	.016	.64258	TMS
3.264	34986	P8	.016	2.94358	
3.369	37314	P8	.015	3.14666	MEMOLA
3.513	78102	P8	.016	6.58626	MeOH
5.078	174858	P8	.015	14.74558	Product
5.686	853034	P8	.018	71.93512	Product

TOTAL AREA: 1185033

MUL FACTOR: 1.0000E+00

DATA

DATA BY

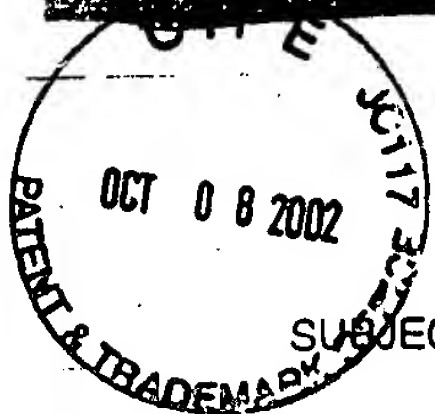
DATE

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CROSS REFERENCES

Original GC in Analytical Results Folder



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DATE

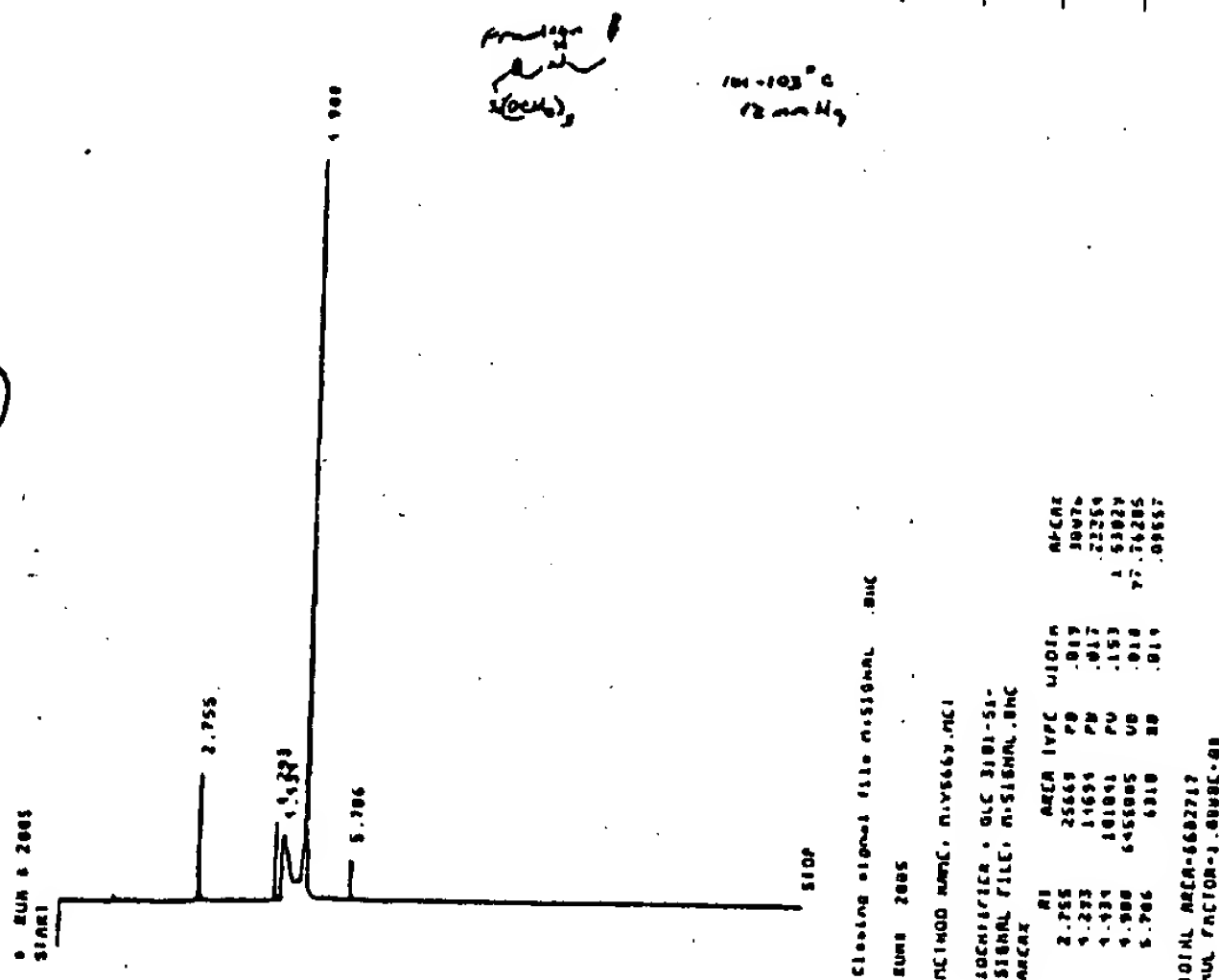
SUBJECT Cont'd PD-3196-8

TEST NO. 3196-11

Time	Temp	Comments
1:05	25	Vacuum @ 23 mm Hg. Heating to 90°C
1:23		Broke vacuum to remove N ₂ inlet. Vacuum ~ 50.
1:27	83/37	Vacuum @ 10 mm Hg.
1:31	85/33	No more lites. Broke vacuum & removed Receiver.
		Replaced w/ 200 mL receiver. Put on ice dry ice/ice.
1:35	90/31	Heating to 120°C. Vacuum @ 9 mm Hg. No sparge.
1:45	102/29	Starting to bubble. " " 14 mm Hg
1:53	104/100	Distilling rapidly. Insulated flask. Vacuum @ 12 mm Hg.
2:03	103/101	" " Vacuum @ 11 mm Hg.
2:24	106/94	Vacuum @ 14 mm Hg.
2:31	105/103	" " 12 mm Hg. Distilling over.
2:48	98/96	" " 9 mm Hg.
3:00	103.3	" " 9 mm. Broke vacuum. Cooling.

Cut 1 = 186.43 g Cut 2 = 86.79 g = 273.22 g total grams

Fraction
#1
of
Distillation



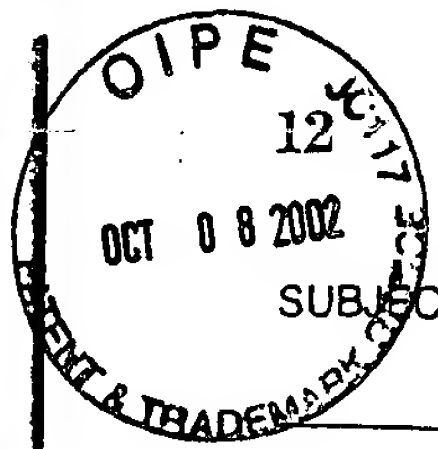
82% yield
isolated

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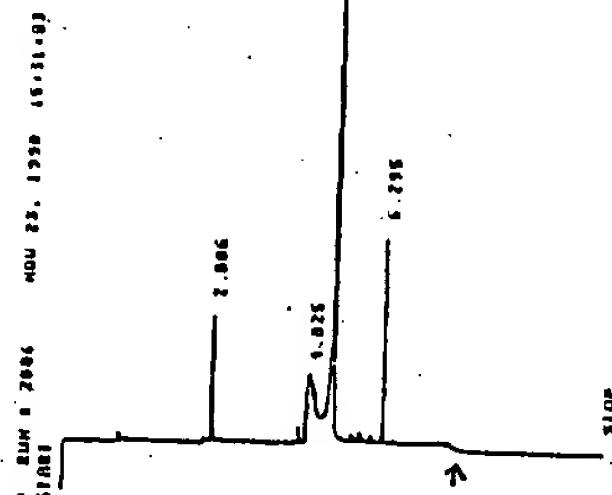
TEST NO. 3196-12

Cont'd PD-3196-8

There was
a front inlet
pressure shutdown
on this last 3
thru it - ok

90 ahead
and mix
w/ later 01

2nd
inlet
with
silicon



Chromatogram data table:

RT	AREA	TYPE	WIDTH	HEIGHT	AREA%
2.886	20885	PU	0.23	15023	1.025
3.825	100024	PU	0.57	117030	5.300
5.295	700001	UB	0.21	271265	6.295
	31211	PU	0.16	11775	

TOTAL AREA=7251978
MUL FACTOR=1.0000E-06

Distillation Cut #2

Mixture of Fractions
1 & 2

11/23/99 15:11:07

STOP

Chromatogram data table:

RT	AREA	TYPE	WIDTH	HEIGHT	AREA%
2.756	33614	PU	0.19	14964	4.496
4.296	12664	PU	0.17	10664	1.664
4.555	107031	PU	0.21	117030	5.300
5.295	700001	UB	0.10	271265	6.295
5.794	10527	PU	0.14	13055	

TOTAL AREA=7624150
MUL FACTOR=1.0000E-06

DATA BY

Beth C. Gaylor

DATE

WITNESSED AND UNDERSTOOD BY

Cathy L. Clegg

DATE

CROSS REFERENCES

Original GC Analysis Results Folder

54

OCT 08 2002

WITCO CORPORATION
OSI SPECIALTIES GROUP
TARRYTOWN, NEW YORK

SUBJECT

Scale Up of γ -1205 & Nemala
(γ -15230)

DATE

TEST NO. PD-3219-SA

1 Obs. Large scale hydrosilation of γ -1205 & N-ethylmethallyl-
2 amine by using M* M*. Sample to be sent to Tarrytown
3 for evaluation.
4

5 Materials: γ -1205 381.31g (5% excess after 97% purity)
6 Nemala 260g
7 M* M* 0.65 mL (50 ppm)
8

9 Equipment: 4 neck ~~fl~~ flask equipped w/ N₂ blowby, addition
10 funnel, distillation apparatus w/ cold finger, thermocouple &
11 magnetic stirrer.
12

13 Procedure: Heat γ -1205 & M* M* to 90°C & drip in Nemala.
14 Heat to 110°C & hold 1 hr.
15

16 Time Temp Comments

17 1:00 Heating to 90°C.

18 1:30 99.7 Dripping in Nemala.

19 1:20 101.9 "

20 1:30 101.6 "

21 2:12 112.9 "

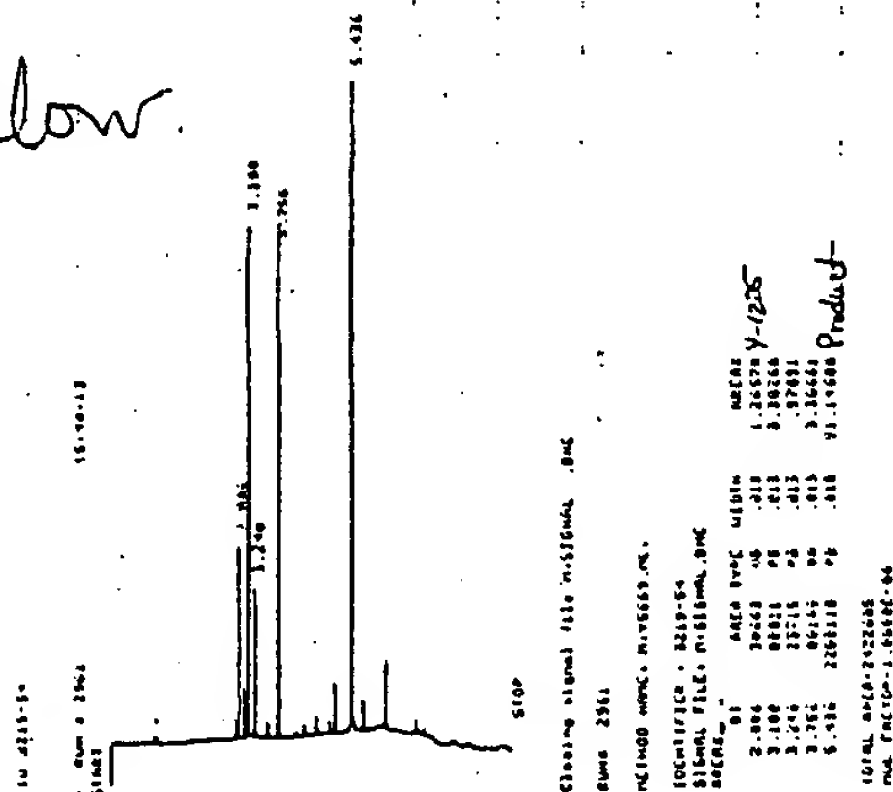
22 2:32 103.0

23 2:42 98.6 All Nemala added. Heating to 110°C. Hold until 3:30

24 3:00 119.8

25 3:30 Cooling to take H₂. Shown Below.

27 By GC, Nemala RT is 3.101



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DATA BY

Beth A. Kaye

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TARRYTOWN, NEW YORK

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Contd PD-3219-54

DATE

TEST NO PD-3219-55

Time	Temp	Comments (Lites Strip)	
8:55		Vacuum @ 26 mm Hg. Heating to 50°C.	1
9:12	41.9	Head temp = 25°C. A few drops coming over.	2
9:18	63.6	HT = 28°C	3
9:37	84.1	HT = 34°C. Very few drops coming over.	4
9:45	94.5	HT = 37°C. Lites are @ 1 drop per 7 seconds.	5
10:14	98.5	HT = 36°C. Lites are @ 1 drop per 14 seconds.	6
10:20	106.4	HT = 50°C. Lites are @ 1 drop per 5 seconds.	7
10:35	116.0	HT = 57°C. " " " " " "	8
10:54	122.4	HT = 60°C. " " " " " " 3 seconds. Vacuum @ 30 mm Hg.	9
11:13	126.4	HT = 68°C.	10
11:47	128.5	HT = 64°C. Cooling to 100°C over lunch.	11
11:55			12
12:40		Heating to 150°C.	13
1:12		Lowered vacuum & removed lites. Temp ~ 132°C. Pulling vacuum & heat.	14
		Lites GC	15
1:19	130.8	HT = 80°C.	16
1:38	129.8	HT = 90°C.	17
2:15	130.1	HT = 90°C.	18
2:30	130.2	HT = 88°C.	19
4:00	130.6	HT = 88°C. vacuum 27 mm Hg. Shut down distillation. placed N ₂ blanket on apparatus. S.C.	20

RAM
01-06-99

DATA BY

DATE

WITNESSED AND UNDERSTOOD BY

DATE

56

OCT 08 2002

WITCO CORPORATION
OSI SPECIALTIES GROUP
TARRYTOWN, NEW YORK

SUBJECT

Conf'd PD-3219-SA

DATE

TEST NO. PD-3219-96

1 Time Temp Comments
 2 8:15 Emptied receiver - collected 150.48 g. Product GC'd.
 3 8:20 Vacuum @ 30 mm Hg. Heating to 150°C.
 4 8:48 132.8 Boiling HT = 72°. Beginning to distill.
 5 9:05 129.8 HT = 102°C. Distilling @ 1 drop per 2 seconds.
 6 9:30 129.8 HT = 98°C. Wrapped w/ foil. Distilling rapidly.
 7 HT = 118°C. Product went to NUS ???
 8 10:01 134.4 HT = 127°C.
 9 10:05 Turned off heat. Very little left in flask.
 10 10:45 GC of receiver. 100% product: same as cut #1.
 11 Collected 328.07g in second cut.
 12
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 33

Total Product distilled = 484.55g

$$\frac{484.55 \text{ g}}{231} = 2.098 \text{ moles}$$

$$\frac{2.098 \text{ g-15230}}{2.626 \text{ NEMANA}} \times 100 = 79.89\% \text{ Isolated yield}$$

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Closing signal file M1SIGNAL.BIN

RUN# 2965

METHOD NAME: M1V5869.MET

IDENTIFIER: 3219-55 LITE

SIGNAL FILE: M1SIGNAL.BIN

APCAR

PI	APCAR	TIME	HEIGHT	AREA
3.759	105965	00	014	5.64497 Y-1205 Impurity
4.590	121087	00	014	6.48251 Cyclic ??
5.044	16196	00	013	0.00379
5.432	1635310	00	017	87.00973 Product

TOTAL APCAR=1877158
MUL FACTOR=1.00000000

DATA BY

Keith D. Kaper

DA

WITNESSED AND UNDERSTOOD BY

Cathy L. Clegg

PATENT & TRADEMARK OFFICE
OCT 08 2002

WITCO CORPORATION
OSI SPECIALTIES GROUP
TARRYTOWN, NEW YORK

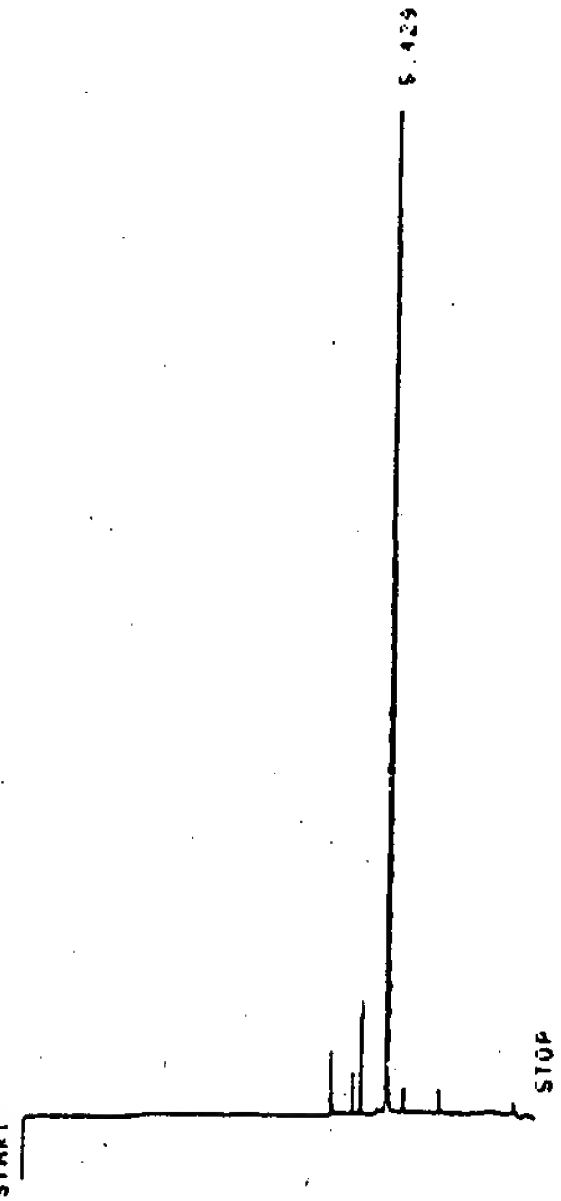
57

SUBJECT WITCO PO-3219-5A

DAT.

TEST NO. PO-3219-57

* RUN # 2973 MAR 31, 1995 00:35:18
START



Closing signal file M: SIGNAL .BNC

RUN# 2973

METHOD NAME: M:V5669.MET

IDENTIFIER: 3219-56-2 PR

SIGNAL FILE: M: SIGNAL .BNC

AREA#

RT AREA TYPE WIDTH AREA#

5.429 1950603 PB 016 100.00000

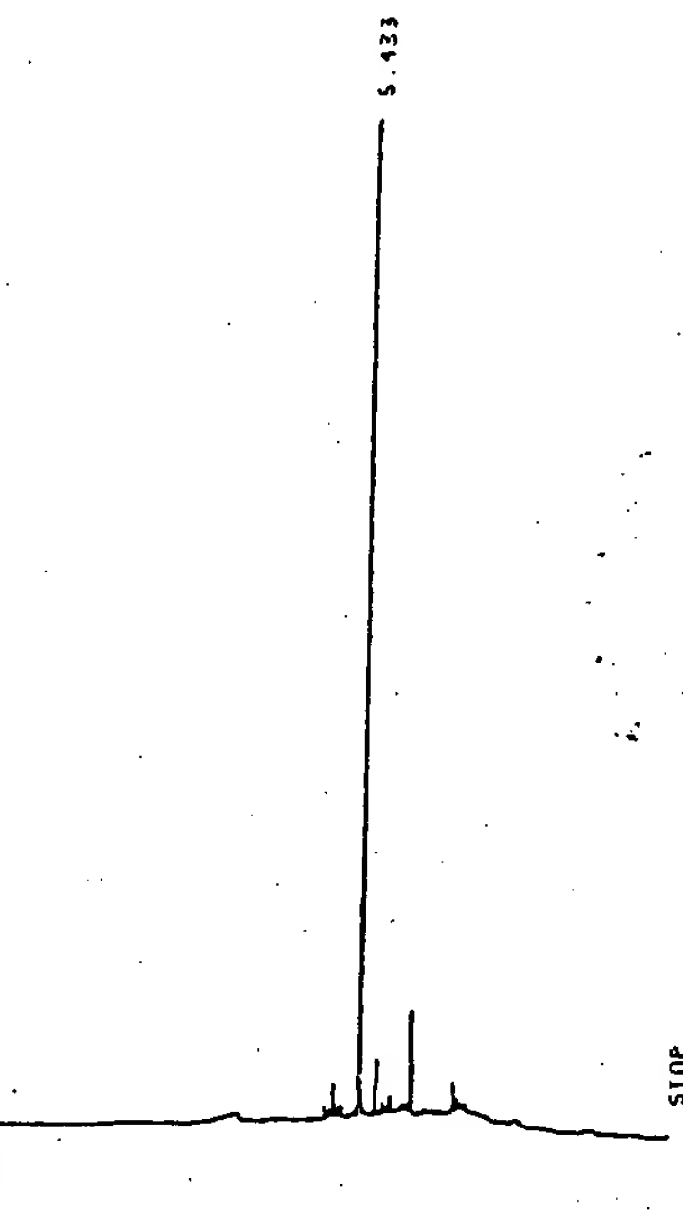
TOTAL AREA=1950603

MUL FACTOR=1.0000E-00

* ID 3219-56-10 2ND PROD. CUI

* RUN # 2978

START



Closing signal file M: SIGNAL .BNC

RUN# 2978

METHOD NAME: M:V5669.MET

IDENTIFIER: 3219-56-10 2

SIGNAL FILE: M: SIGNAL .BNC

AREA#

RT AREA TYPE WIDTH AREA#

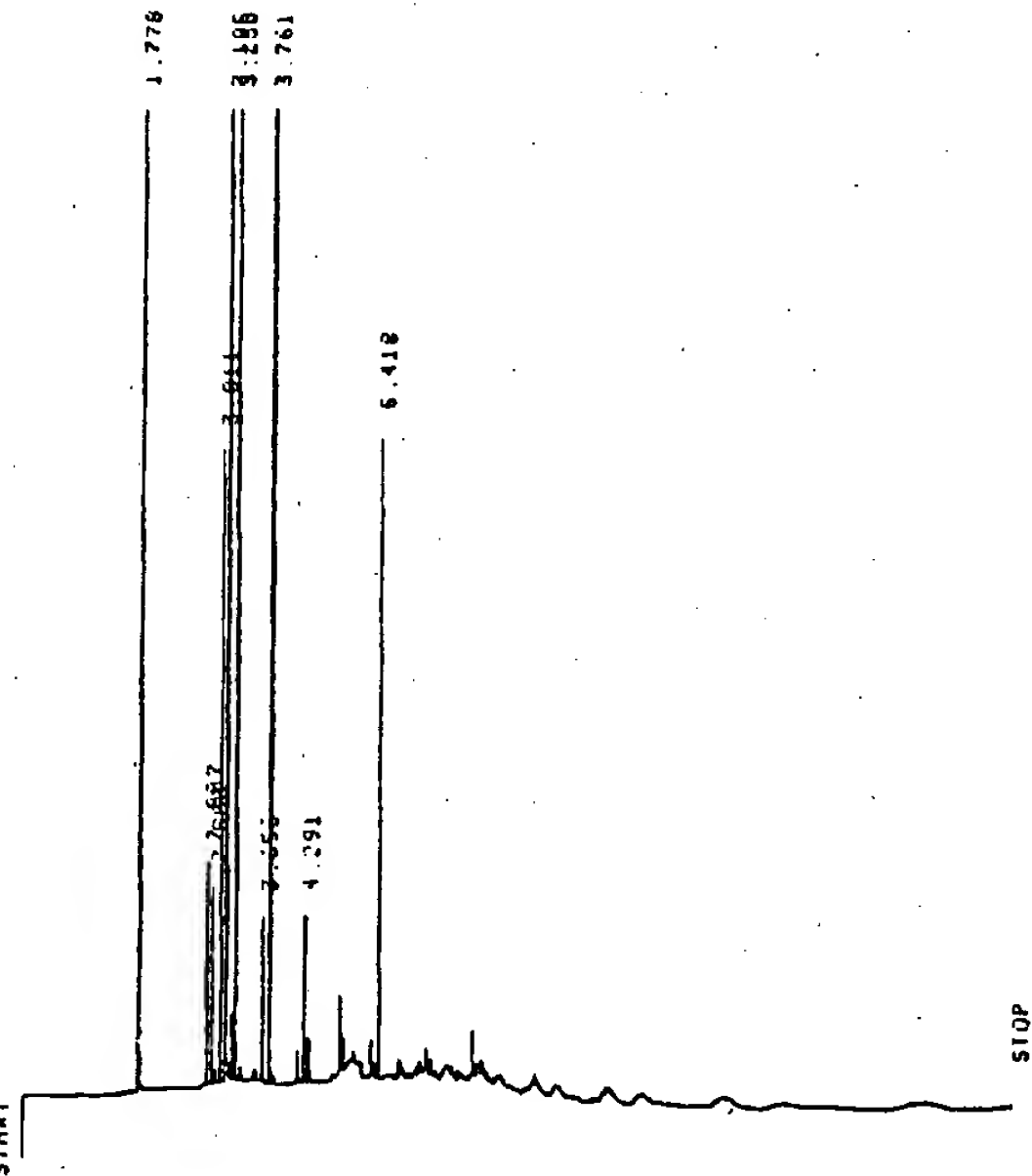
5.433 1950470 PB 016 100.00000

TOTAL AREA=1950470

MUL FACTOR=1.0000E-00

* ID MAT'L FROM TRAP

* RUN # 2985 MAR 31, 1995 13:51:46
START



Closing signal file M: SIGNAL .BNC

RUN# 2985

METHOD NAME: M:V5669.MET

IDENTIFIER: MAT'L FROM 1

SIGNAL FILE: M: SIGNAL .BNC

AREA# RT AREA TYPE WIDTH AREA#

1.778 189827 PB 014 6.95432

2.807 23625 PB 013 1.45595

3.011 65702 PB 012 1.29829

3.105 552100 PB 012 4.15661

3.250 157725 PB 013 34.96493

3.656 24740 PB 010 5.90725

3.761 541707 PB 014 1.56655

4.291 17307 PB 013 24.36125

5.418 65900 PB 015 1.10896

5.418 65900 PB 015 4.17790

TOTAL AREA=1579263

MUL FACTOR=1.0000E-00

DATA BY

Beth A. Lopez

DA

WITNESSED AND UNDERSTOOD BY

Cathy L. Clegg

DA